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ABSTRACT

Fiber-optic sensors are mostly used for in situ measurements of diverse chemical composition of industrial surfactants employed in industry as detergents, emulsifying and dispersing agents, coatings, and pharmaceutical adjuvants. These optical sensors are often used in wet chemical environments in which the temperature can be high.

The purpose of this work is to study the mechanical behavior of optical fibers in contact with CetylTrimethylAmmonium Chloride in aqueous solution (CTAC) at different immersion durations and different temperatures.

Result analysis demonstrates that immersion in CTAC drastically decreases the fiber strength particularly when immersed for long aging periods at high temperatures.

Based on the analysis of aged fiber surface morphology obtained from Scanning Electron Microscopy, the extent of the damage of the fiber core and polymer coatings was observed.

1. Introduction

Surfactants (Surface Active Agents) are used as detergents, dispersants or pharmaceutical adjuvants. In some case, surfactants can play a vital role when used for health issues, for example a pulmonary surfactant is a mixture of lipids and proteins that covers alveolar collapse during respiration [6,8,31,33].

A surfactant modifies the surface tension between two surfaces and normally contain both one hydrophilic (water soluble) group and one hydrophobic (oil soluble) chain [1–3].

Surfactants can be classified as follows: anionic, amphoteric, nonionic or cationic surfactants. This depends on the different functions of the surfactant [16].

One of the characteristic properties of these substances is their capacity to aggregate in aqueous solutions above a certain concentration known as the Critical Micelle Concentration CMC [10].

This relies on the fact that a solution containing surfactants presents high changes at CMC. These changes affect the physical and chemical solution properties such as electrical conductivity, surface tension, and detergent activity. At the CMC point, the water surface tension is reduced by the surfactant which adsorbs the liquid–gas interface. Above this point, stable aggregates are spontaneously formed.

For cleaning industries, it is important for economical reasons to find the CMC point because the detergent activity does not effectively change after this point. Many measurements have been made with water as a solvent at different surfactant concentrations near the CMC point, using different techniques like optical fiber sensors [24–26,30].

Based on the measurement of evanescence wave adsorption [5,7,14,17,23,28,29], optical fiber sensors are increasingly used. There is a growing interest in this method due to the fact that there is fast implantation of optical fiber probes, which are adapted for in situ measurements, and there is no need of a reference electrode or several samples [7]. Optical fiber probes lead to several advantages as rugged construction, and long interaction length. Fiber sensors can measure the refractive index of the surfactant solutions using a two-channel Fresnel reflexion technique [30]. To detect the CMC point, the optical fiber optically transmits a wave which interacts with the surfactant molecules at the interface and passes trough the sensing region along the fiber with repeating reflections [25].

But near the CMC point, surfactants adsorb at solid/water interfaces (particularly at the surface of hydrophilic oxide of silica fiber) and lead to a significant decrease of the mechanical fiber

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structure as the fiber strength and the polymer coating can be seriously damaged.

El Abdi et al. [11,12] have studied the effect of different surfactants on the strength of silica optical fibers. But fibers were aged during small aging durations and in ambient temperature.

Using a dynamic two point bending set-up, the evolution of mechanical properties of optical fibers versus water temperature, immersion duration, and dynamic test velocity was analyzed for aging in a cationic surfactant solution at CMC concentration.

2. Experimental

2.1. Surfactant used

Cetyltrimethylammonium chloride solution (CAS number 112-02-07) is a cationic surfactant used as a very toxic antiseptic but can also be used as a phase-transfer catalyst under conditions which prevent emulsions. The adsorption of this surfactant on solid/liquid interfaces is important in different processes. The analysis of this adsorption helps to understand the biological phenomena, the detergent effects and the control of pollution.

This cationic surfactant, belonging to a class of quaternary ammonium salts, is a known compound and offers some additional advantages over other surfactants [9,15,18,27].

The toxicity of CTAC for aquatic bacteria is often used in industrial cleaning up. CTAC is regarded as a cationic softener, lubricant, retarding and antistatic agent and, in some cases, is used for consumer use [21].

CTAC was purchased from Sigma Aldrich Co. (France) (25 wt. % in H_2O).

Table 1 gives CTAC properties at different temperatures and detailed formula.

2.2. Optical fiber and adiabatic enclosure used

Heavily protected optical fibers are designed for use at elevated temperatures and pressures in aggressive chemical environments.

Because the high cost and due to the great test number, only one type of optical fibers was studied. The used multimode fiber has two acrylate coatings (primary and outer coatings) (Fig. 1). This fiber has a numerical aperture of 0.2 (NA value) with an operating wavelength of 850/1300 nm. A soft, primary coating has a low module of elasticity, adheres closely to the glass fiber and forms a stable interface. It protects the fragile glass fiber against micro-bending and attenuation. The outer coating protects the primary coating against mechanical damage and acts as a barrier to lateral forces. It has a high glass transition temperature and Young modulus. It has good chemical resistance and serves as a barrier against moisture. The combined coating diameter is 245 μm, the silica core has a diameter of 50 μm and the clad diameter is 125 μm (Fig. 1).

Before the dynamic bending tests, fibers are plunged into a container with a distilled water-surfactant solution at CMC concentration. This container itself is then deposited in water at different temperatures. An adiabatic enclosure maintains a constant temperature during the aging period (Fig. 2). For three different temperatures (ambient temperature, 30 °C and 60 °C), optical fibers were aged in hot water at a CMC concentration given in Table 1, during one, two or three weeks with a relative humidity of between 35 and 45%.

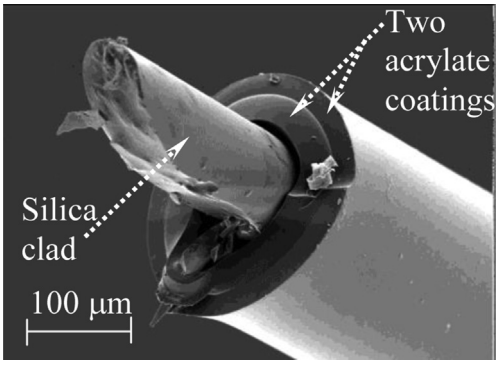

Fig. 1. Used silica optical fiber.

2.3. Bending test bench

While the bending method does not replace tensile testing as a fiber strength measurement technique (the tensile test rapidly provides several material characteristics such as Young's modulus, elastic limit, Poisson's coefficient, tensile strength, break elongation…), it presents attractive features and advantages, providing valuable information about flaw size distribution [22]. In our case, the ease and the duration of the testing manipulations and the small effective length of the fiber sample made the bending test the most appropriate choice for investigation.

The 'as-received' fibers and those aged in CTAC solution were put subsequently through dynamic tests using a two-point bending testing device (Fig. 3). The fiber package was cut into two 8 cm length parts.

Special care was required to avoid the fiber slipping during the faceplate displacement and to maintain the fiber ends in the same vertical plan.

The two points bending bench is made up of a displacement plate which is mounted on an aluminum plate (Fig. 3a). The first thrust block is movable and mounted on the displacement plate, while the second thrust block is fixed on a force sensor. The optical fiber is positioned between the two thrust blocks in such a way that it forms a "U". To avoid slipping, the fiber is positioned in the grooves of the thrust blocks (Fig. 3b and c).

During the test, load and displacement are recorded, allowing the load/displacement curve to be obtained. At breaking point, the stress applied to the fiber was deduced using the distance d between the two faceplates (Fig. 3c). A non linear relation defined by Proctor and improved by Griffioen [13] can give the evolution of the stress σ (GPa) as a function of second polynomial order i.e.:

$$\sigma = E_0 \cdot \varepsilon \left(1 + \frac{\alpha'' \cdot \varepsilon}{2}\right) \quad (1)$$

Table 1
Physical and chemical CTAC properties.

Product	Molecular weight (g/mol.)	pH at 20 °C concentration: 20 g/L	Boiling point (°C)	Density (g/cm^3)	CMC[a] (mmol./L)
Cetyltrimethylammonium chloride solution (CTAC) $C_{19}H_{42}ClN$	320	6–7	100	0.968	1.35 at 20 °C 1.40 at 30 °C 1.62 at 60 °C

[a] CMC: Critical micellar concentration.

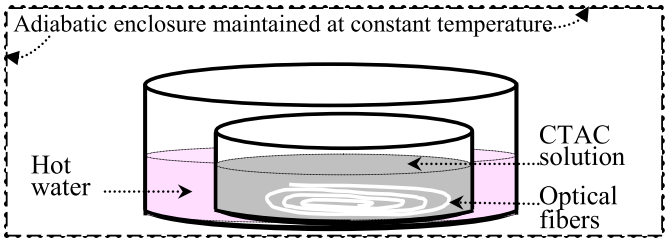

Fig. 2. Adiabatic enclosure for aging of optical fibers.

where the first term describes the linear contribution (Hooke's law) and the second term accounts for a quadratic contribution. This law is often used for elastic or elastic–plastic behavior with low plasticity as in the case of the used optical fibers.

In Eq. (1),

$$\alpha'' = \frac{3}{4}\alpha + \frac{1}{4} \tag{2}$$

where α is a non linear elastic parameter (typical value of α is 6).

The strain ε of the fiber depends on the core diameter of the fiber (the most rigid part of the fiber) and of the distance between the two branches at rupture. ε was defined by:

$$\varepsilon = 1.198 \left[\frac{d_f}{d - d_c + 2d_g}\right] \tag{3}$$

where E_0 is the Young modulus equal to 72 GPa for silica, d_c is the polymer coating diameter, d_f is the fiber diameter, $2d_g$ is the total depth of the 2 grooves and d is the distance between the two faceplates (Fig. 3c).

Initially, the distance between the two plates (fixed block and movable plate), between which the optical fiber is placed, is 10 mm. The movable plate moves to the left and thereby compresses the fiber which breaks in its middle. Fig. 4 shows a typical force–displacement curve. One can note that the load gradually increases when the movable plate moves closer to the fixed block and the load decreases drastically after the fiber breaks. The distance d between the two parts of the fiber at rupture is equal to: 10 mm minus the distance covered by the movable plate before fiber breaking.

In the case of Fig. 4, this rupture distance will be 2.45 mm (10 mm–7.55 mm). Using Eq. (1), the failure stress σ was equal to 3.7 GPa.

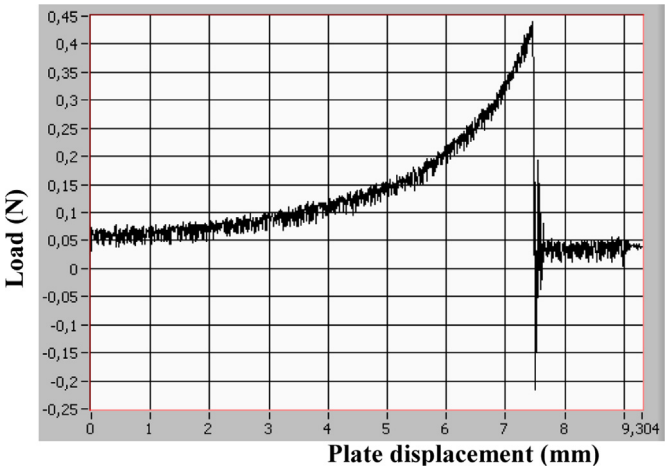

Fig. 4. Load–displacement curve for an optical fiber aged during 3 weeks in CTAC solution at 60 °C (movable plate velocity = 0.8 mm/s).

3. Results and discussion

'As-received' optical fibers (as reference) as well as fibers immersed in distilled water mixed with CTAC at different temperatures were aged for different durations, and then dried for 3 days before the bending tests for 5 velocities of the movable faceplate: 0.8, 2.6, 4.4, 6.2 and 8 mm/s. For each velocity, the test was carried out on 10 fibers and the mean value was selected as final value.

Fig. 5 shows the distance d between the two faceplates (Fig. 3c) when the fiber breaks and the failure load for different faceplate velocities for the fiber aged in CTAC solution at room temperature (21 ± 2 °C).

The distance slowly increases (Fig. 5a) and the failure load decreases (Fig. 5b) when the aging period increases.

Immersion in the CTAC solution damages the optical fiber, which breaks faster as the immersion duration is greater. For that reason, the failure distance d is higher as the immersion time is greater and the breaking strength decreases. This phenomenon was even more pronounced when the solution temperature increases (Fig. 6). The failure distance increased (about 17% for immersion of one week in comparison for the aging at room temperature). The decrease in the breaking strength is drastic (loss of 45% between the values of the 'as received' fiber and that of the fiber aged for 2 weeks) and stabilized after 2 weeks (Fig. 6b). A high faceplate velocity (8 mm/s) compresses the fiber rapidly and then leads to a breaking strength greater than that obtained for a low velocity (0.8 mm/s). One can note that for a long aging period, the force curves will join to the lower breaking strength.

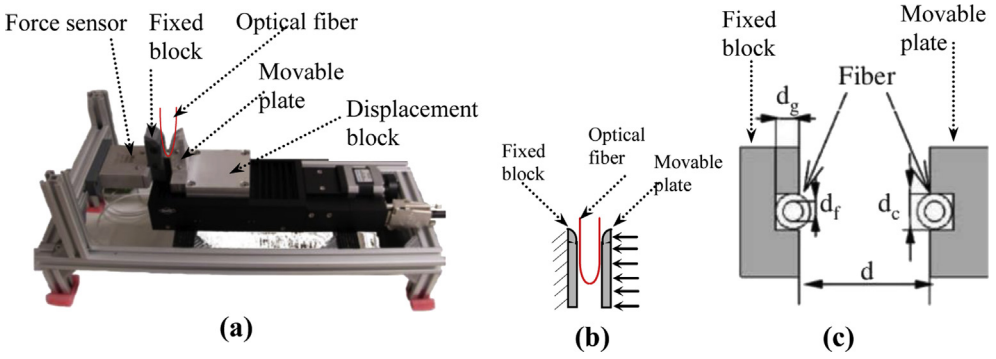

Fig. 3. (a) Bending bench used; (b) and (c) fiber between thrust blocks.

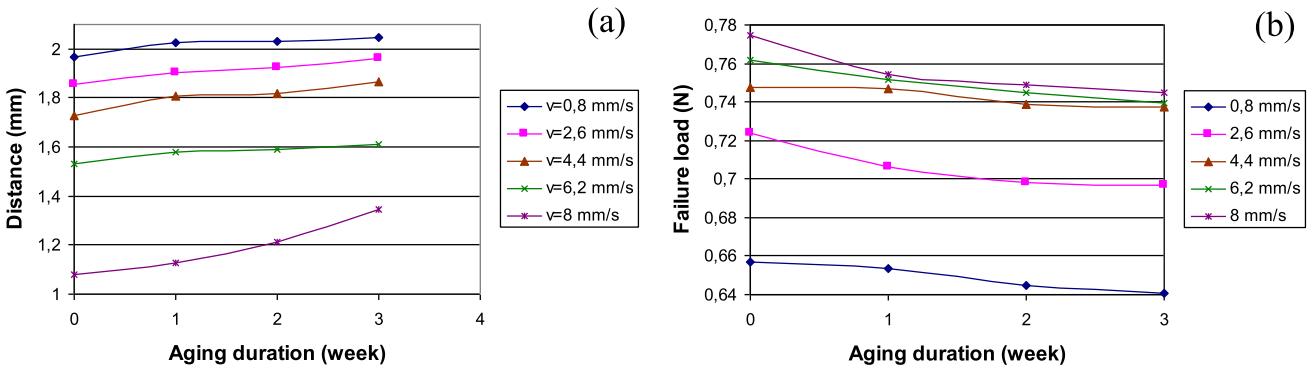

Fig. 5. Failure distance and load at room temperature for different faceplate velocities.

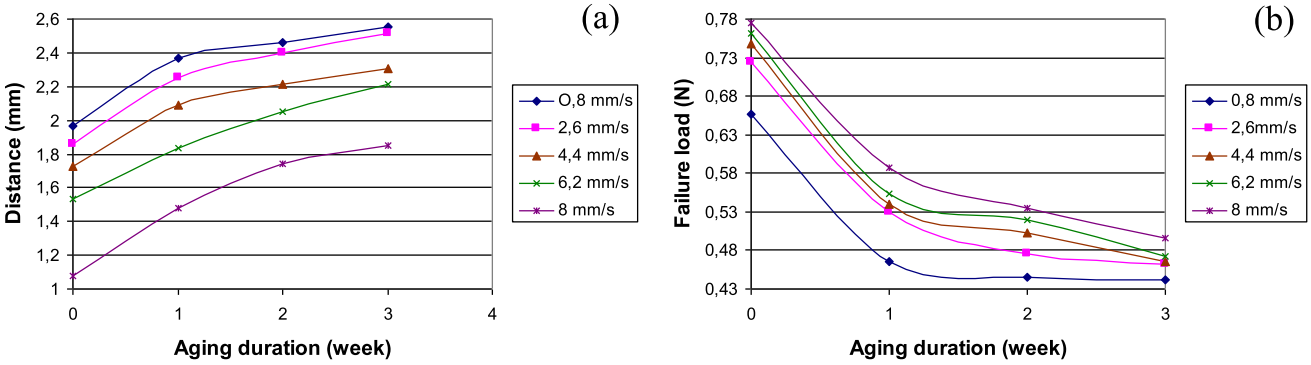

Fig. 6. Failure distance and load for aging temperature of 60 °C for different faceplate velocities.

For a moderate aging temperature (30 °C) (Fig. 7), the aging effect is slightly pronounced. For 'as-received' fibers and for three aging durations, the obtained curves were not very distant from each other. An aging temperature equal to or greater than 60 °C was needed to obtain significant fiber damage. At 60 °C, the loss in fiber resistance was about 35% compared to that of 'as-received' fibers (Fig. 8b) and this loss is high especially for longer aging periods. Fig. 9 shows the loss of strength for an immersion of 3 weeks and the large amount of damage obtained at 60 °C.

4. SEM observations

An overall image with detail of the optical fibers is given in Figs. 10 and 11.

The details given by the Scanning Electron Microscope corroborate the experimental results obtained by bending tests. Indeed, Fig. 10 shows the 'as-received' fiber submitted to two faceplate velocities: low velocity (0.8 mm/s) and high velocity (8 mm/s). For a low faceplate velocity (Fig. 10a), the crack propagation is not perpendicular to the fiber axis but the micro-crack is propagated

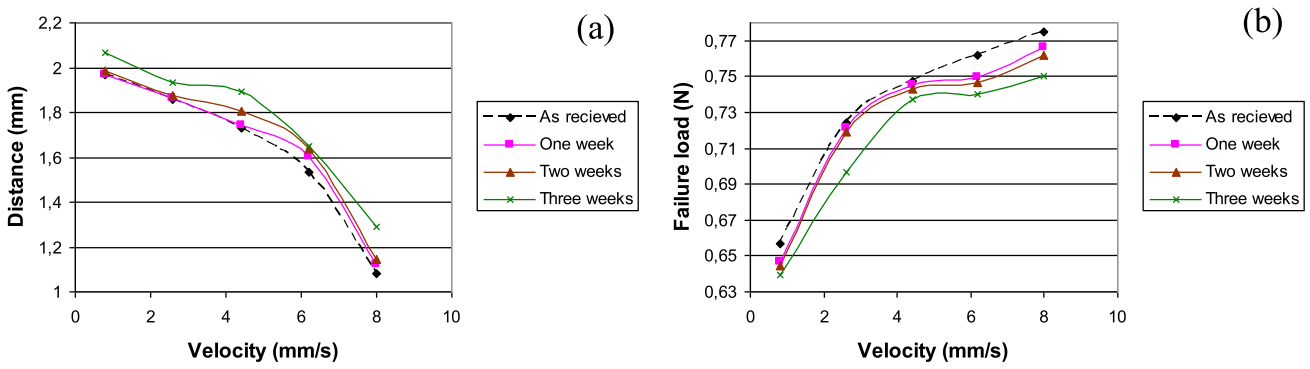

Fig. 7. Failure distance and load for aging temperature of 30 °C for different aging durations.

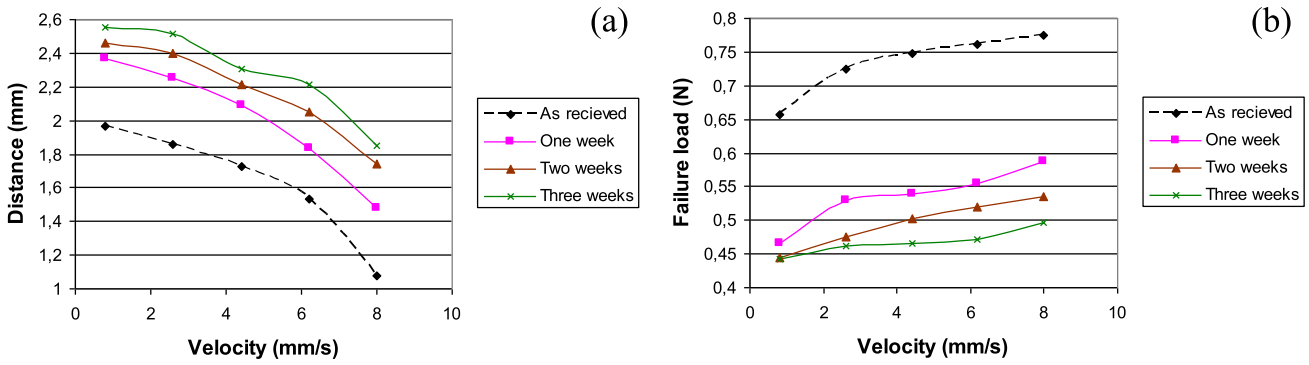

Fig. 8. Failure distance and load for aging temperature of 60 °C for different aging durations.

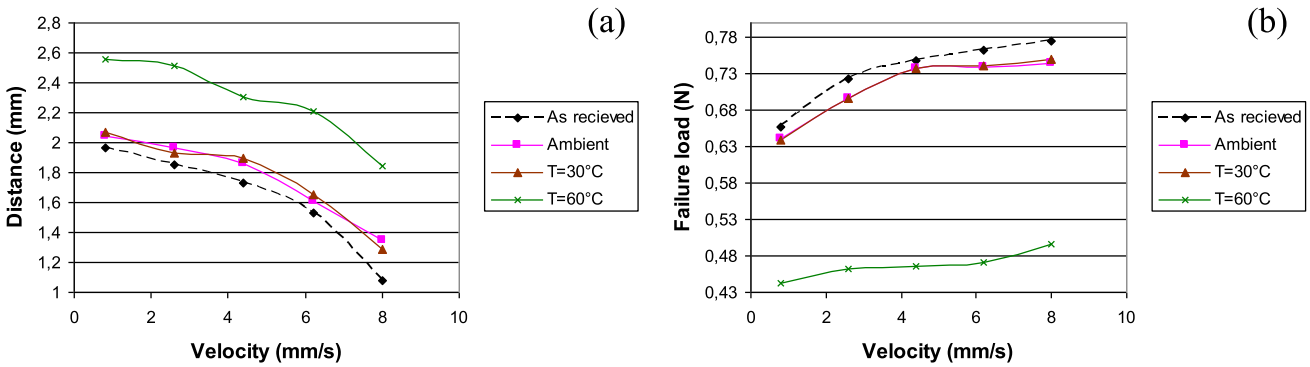

Fig. 9. Failure distance and load for aging duration of 3 weeks for different aging temperatures (ambient temperature: 21 ± 2 °C).

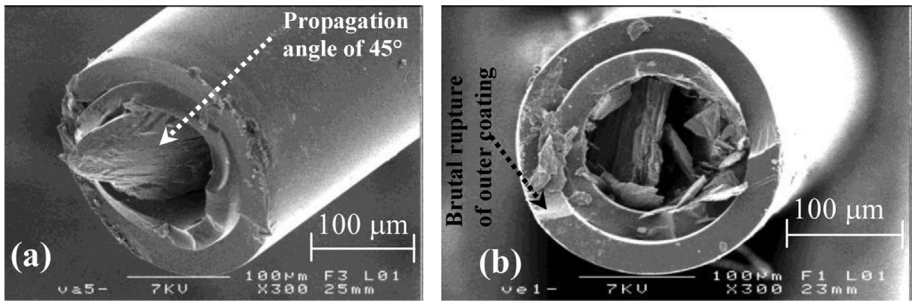

Fig. 10. 'As-received' fibers-faceplate velocity: (a) 0.8 mm/s and (b) 8 mm/s.

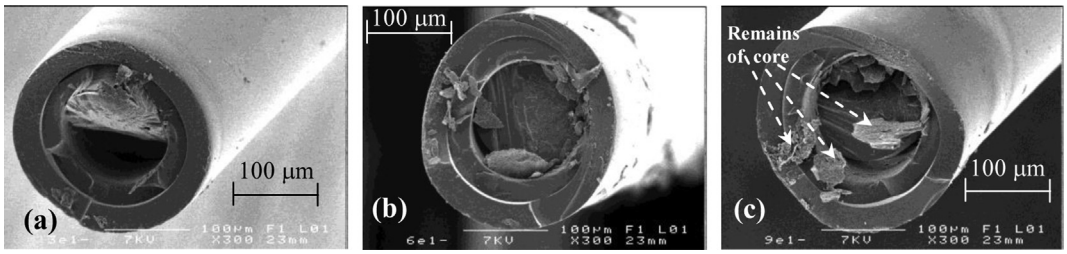

Fig. 11. Three week aged fibers at: (a) ambient temperature, (b) 30 °C, (c) 60 °C.

with an angle of 45° (in the case of bending test, the fiber was bent into a "U" shape. The internal surface of the fiber (small curvature radius) was submitted to the compressive stresses and its external surface was submitted to tensile stresses. These stresses normal to the fiber section lead to a propagation angle of 45 °C and the propagation track was a straight line as silica was a brittle material). For a high faceplate velocity (Fig.10b), the fiber undergoes rapid deformation that leads to a violent fracture of the fiber core and separation of the primary coating from the outer coating. When the fiber undergoes the bending test, the outer coating is subjected to the highest bending stress which leads to a brutal rupture as indicated in Fig. 10b.

Fig. 11 shows aged broken fibers after bending tests for 8 mm/s after immersion in CTAC solution at different temperatures for 3 weeks.

When the temperature of the CTAC solution is low, the fiber resists aging damage (Fig.11a). When the temperature of the solution increases, the coating is more susceptible to water damage, the core is weakened and breaks into several pieces during the bending test. This is more visible with high temperature (60 °C) (Fig.11c).

The degradation of the silica fiber strength in distilled water is monitored by increasing surface roughness due to the dissolution of silica on the surface of the fiber by water corrosion [19,32]. When a water polar species ruptures the silicon–oxygen bond, dissolution occurs controlled by the following equation:

$$SiO_2 + H_2O \rightarrow Si - O - H \quad H - O - Si \tag{4}$$

Silicon–oxygen bonds are progressively broken, advancing the micro-crack and the fiber is weakened.

If one can segregate the water effect from the silica surface, the fiber strength does not present a notable decrease (perhaps a minor decrease can be observed and will be due to the residual moisture inserted between the silica surface and the polymer during the coating application). The fiber strength change after aging depends, thus, on the permeability of the used coatings, only hermetic coatings are considered capable of completely preventing water from reaching the glass surface [4] and the used acrylate coating shows a small permeability to water diffusion.

On the other hand, surfactant molecules comprise heads and tails. Heads are hydrophilic components and tails are hydrophobic components. For cationic surfactants such as CTAC solutions, the hydrophilic part is positively charged and releases a positive charge (cation) in aqueous solution.

The hydrophilic groups of the CTAC molecules dissolve in water before adsorption onto the silica surface which comprises hydrophilic hydroxide groups OH (Eq. (4)) and onto the hydrophilic polymer coating. When the concentration is below the CMC point, surfactant molecules are scattered in the solution and a small adsorption is initiated onto the optical fiber and molecular hydrophobic parts are attracted onto the surface of the interface between the air and the surfactant solution. At the CMC, all the surfaces of optical fiber were covered with monolayers of surfactant molecules and the surfactant action was maximal. As shown in Table 1, the critical concentration increases with the temperature.

The combined effect of this critical concentration, water and temperature leads to severe damage of the optical fiber.

5. Conclusion

Based on the measurement of evanescence wave adsorption, sensors with optical fibers were used in situ to measure solution concentration with surfactants. These optical sensors are sometimes used at different temperatures and can be damaged if the immersion time is long enough.

The mechanical behavior of fibers immersed in Cetytrimethylammonium chloride solution was analyzed. The experimental results illustrate the change of the strength and the rupture fiber curvature for high temperature and for long immersion periods. That was confirmed by the SEM observations.

The immersion duration in CTAC solution affected the fiber's mechanical strength. But it was especially the temperature which increased damage and ruptures and decreased fiber strength.

Up to a temperature of 30 °C, the mechanical strength of the fiber decreases but it is still acceptable. Beyond 30 °C and at about 60 °C, the decrease in resistance is significant and leads to significant fiber damage mainly due to the core having a high elasticity modulus compared to that of the polymer coatings.

The polymeric coating resists water attack, but a CTAC solution at 60 °C weakens the coatings. The faceplate velocity also plays a significant role and a high bending velocity leads to a rapid fiber breakdown (for a high bending velocity, a rapid fiber breakdown was obtained but the distance covered by the movable plate was great and thus the distance d between the branches was small) and to a high breaking load.

Finally, for a short aging period and a not high temperature, the multimode optical fibers can be used. But for long immersion periods and for a temperature higher than 40 °C, the use of hermetic optical fibers is advised. Such fibers are designed to improve aging behavior and to avoid diffusion through the glass surface and coating damage.

Lu et al. [20] have examined the synergistic effect of self-assembled carbon nanofiber (CNF) nanopaper and multi-layered interface on the electrical property and electro-activated recovery behavior of shape memory polymers nanocomposites. The surfactant Triton X-100 was used to aid the dispersion of CNF. An improvement of electrical conductivity and heat transfer was obtained.

In our case, the synergistic effect to immersion duration and temperature on the optical properties of fibers can be studied in future work.

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